

9.0 QUALITY ASSURANCE

It is the policy of the U. S. Department of Energy, National Nuclear Security Administration Nevada Operations Office (NNSA/NV) that all data produced for its environmental surveillance and effluent monitoring programs be of known quality. Therefore, a quality assurance (QA) program is used for collection and analysis of samples for radiological parameters to ensure that data produced by the Bechtel Nevada (BN) in-house Analytical Services Laboratory (ASL) and Subcontracted Radiochemistry Laboratory meets customer-and regulatory-defined requirements. Data quality is assured through process-based QA, procedure-specific QA, measurement quality objectives (MQOs), and performance evaluation programs (PEPs). The QA program for radiological data consists of participation in the Quality Assessment Program (QAP) administered by the NNSA/NV Environmental Measurements Laboratory (EML), the InterLaB RadCheM™ Proficiency Testing Program directed by Environmental Resource Associates (ERA), the Radiochemistry Intercomparison Program provided by the National Institute of Standards and Technology (NIST), and the Mixed Analyte Performance Evaluation Program (MAPEP) conducted by the Idaho National Engineering and Environmental Laboratory (INEEL). Thermoluminescent dosimeter (TLD) radiation measurement QA for the program is assessed by the BN Dosimetry Group's participation in the NNSA/NV's Laboratory Accreditation Program and intercomparisons provided by the Battelle Pacific Northwest National Laboratory (PNNL) during the course of the year.

9.1 POLICY

Environmental surveillance, conducted onsite by BN, is governed by the NNSA/NV QA policy as set forth in DOE Order 414.1A (DOE 1999). The Order outlines ten specific elements that must be considered for compliance with the QA policy. These elements are:

1. Program
2. Personnel Training and Qualification
3. Quality Improvement
4. Documents and Records
5. Work Processes
6. Design
7. Procurement
8. Inspection and Acceptance Testing
9. Management Assessment
10. Independent Assessment

9.2 OVERVIEW OF THE LABORATORY QA PROGRAM

The BN in-house Analytical Services Laboratory (ASL) and the Subcontracted Radiochemistry Laboratory implements the requirements of the DOE Order O 414.1A through integrated quality procedures. The quality of data and results is ensured through both process-based and

procedure-specific QA. BN is assured of quality data from the Subcontractor through both a review of the Subcontractor's QA Plan by BN as well as the Subcontractor's successful participation in the NNSA's Environmental Consolidated Audit Program (EMCAP).

Procedure-specific QA begins with the development and implementation of Organizational Procedures (OPs), which contain the analytical procedures and required quality control samples for a given analysis. Personnel performing a given analysis are trained and qualified for that analysis, including the successful analysis of a quality control sample. Analysis-specific operational checks and calibration standards traceable to either the NIST or the U. S. Environmental Protection Agency (EPA) are required. Quality control samples, e.g., spikes, blanks, and replicates, are included for each analytical procedure. Compliance with analytical procedures is measured through procedure-specific assessments or surveillances.

An essential component of process-based QA is data review and verification to assess data usability. Data review requires a systematic, independent review against pre-established criteria to verify that the data are valid for their intended use. Initial data processing is performed by the analyst or health physicist generating the data. An independent review is then performed by another analyst or health physicist to ensure that data processing has been correctly performed and that the reported analytical results correspond to the data acquired and processed. Supervisory review of data is required prior to release of the data to sample management personnel for data verification. Data verification ensures that the reported results correctly represent the sampling and/or analyses performed and includes assessment of quality control sample results. Data processing by sample management personnel ensures that analytical results meet project requirements. Data checks are made by Environmental Technical Services of BN for internal consistency, proper identification, transmittal errors, calculation errors, and transcription errors.

Process-based QA programs also include periodic operational checks of analytical parameters such as reagent water quality and storage temperatures. Periodic calibration is required for all measuring equipment such as analytical balances, analytical weights, and thermometers. The overall effectiveness of the QA program is determined through systematic assessments of analytical activities. Systematic problems are documented and corrective actions tracked through System Deficiency Reports.

Similar procedures and methodologies are used by the Subcontracted Radiochemistry Laboratory to ensure the quality of environmental radiological data they produce.

9.3 MEASUREMENT QUALITY OBJECTIVES (MQOs)

MQOs are commonly described in terms of representativeness, comparability, precision, accuracy, blank analysis, and interlaboratory comparison studies. Definite numerical goals may be set and quantitative assessments performed for these components of the data.

REPRESENTATIVENESS

Representativeness is the degree to which a sample is truly representative of the sampled medium; i.e., the degree to which measured analytical concentrations represent the concentrations in the medium being sampled (Stanley and Verner 1985).

Representativeness also refers to whether the locations and frequency of sampling are such that calculational models will lead to a correct estimate of potential EDE to a member of the public when measured radioactivity concentrations are put into the model. An environmental monitoring plan for the, "Nevada Test Site Routine Radiological Environmental Monitoring Plan" (DOE 1998a) has been established to achieve representativeness for environmental data. Factors which were considered in designing this monitoring plan include locations of known and potential sources, historical and operational knowledge of isotopes and pathways of concern, hydrological, and topographical data, and locations of human populations.

COMPARABILITY

Comparability refers to the degree of confidence and consistency in the laboratory's analytical results, or defined as "the confidence with which one data set can be compared to another" (Stanley and Verner 1985). To achieve comparability in measurement data, sample collection and handling, laboratory analyses, and data analysis and validation are performed in accordance with established OPs. Standard reporting units and a consistent number of significant digits are used. Instruments are calibrated using NIST-traceable sources. Extensive QA measures are used for all analytical processes.

PRECISION

Precision refers to "the degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions" (Taylor 1987). Practically, precision is determined by comparing the results obtained from performing the same analysis on split samples, or on duplicate samples taken at the same time from the same location, maintaining sampling and analytical conditions as nearly identical as possible. Precision for samples is determined by comparing results for duplicate samples of particulates in air, tritiated water vapor, TLDs, and of some types of water samples. Control limits for duplicates have been established at \pm detection level for results less than 5 times detection level. If the result is greater than 5 times detection level, then results must be \pm 20 percent Relative Percent Difference (RPD).

ACCURACY

Accuracy refers to how well we can measure the true value of a given quantity and can be defined as "the degree of agreement of a measured value with the true or expected value of the quantity of concern" (Taylor 1987). For practical purposes, assessments of accuracy for the ASL and Subcontract Radiochemistry Laboratory are done by performing measurements on a Laboratory Control Sample (LCS) which is sometimes called a Blank Spike Sample (BSS). An LCS is a control sample of known composition which is analyzed using the same sample preparation, reagents, and analytical methods as employed for the project samples.

The accuracy of these measurements, which is assumed to extend to other similar measurements performed by the laboratory, may be defined as the ratio of the measured value divided by the true value, expressed as a percent. The control limits (in percent) for accuracy that is monitored by using LCS results, are 80 to 120 percent except for gross alpha and beta which are 50 to 120 percent.

BLANK ANALYSIS

A blank analysis is an artificial sample designed to monitor the introduction of artifacts into the measurement process. There are several types of blanks which monitor a variety of processes:

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- **A laboratory blank** is taken through sample preparation and analysis only. It is a test for contamination in sample preparation and analyses.
 - **A holding blank** is stored and analyzed with samples at the laboratory. It is a test for contamination in sample storage as well as sample preparation and analysis.
 - **A trip blank** is shipped to and from the field with the sample containers. It is not opened in the field and, therefore, provides a test for contamination from sample preservation, site conditions, and transport as well as sample storage, preparation, and analysis.
 - **A field blank** is opened in the field and tests for contamination from the atmosphere as well as from sample preservation, site conditions, transport, sample storage, preparation, and analysis.

For the BN Environmental Monitoring Program laboratory blanks are monitored, with a control limit of less than the detection level being used.

INTERLABORATORY COMPARISON STUDIES

The BN in-house Analytical Services Laboratory (ASL) and Subcontracted Radiochemistry Laboratory analyze special QA samples that are prepared, using stringent quality control, by laboratories which specialize in preparing such samples. The values of the activities of these samples are not known by the staff of the ASL or the Subcontracted Laboratory until several months after the measurements are made and the results sent back to the QA laboratory. These sample values are unknown to the analysts and serve to measure the capability of a laboratory for analyzing an analyte in a specific matrix.

The interlaboratory comparison studies that the ASL and Subcontracted Radiochemistry Laboratory participate in are the Quality Assessment Program (QAP) administered by the NNSA Environmental Measurements Laboratory (EML), the InterLaB RadCheM™ Proficiency Testing Program directed by Environmental Resource Associates (ERA), the Radiochemistry Intercomparison Program provided by the National Institute of Standards and Technology (NIST), and the Mixed Analyte Performance Evaluation Program (MAPEP) conducted by the Idaho National Engineering and Environmental Laboratory (INEEL).

The capability of the BN Dosimetry Group's TLD program is tested during the course of the year by their participation in the Battelle Pacific Northwest National Laboratory (PNNL) performance evaluation study program. They are also tested every two or three years by the NNSA's Laboratory Accreditation Program. This involves a three-part, single blind performance testing program followed by an independent onsite assessment of the overall program.

9.4 RESULTS FOR DUPLICATES, LABORATORY CONTROL SAMPLES, BLANK ANALYSIS, AND INTERLABORATORY COMPARISON STUDIES

A brief discussion of the year 2000 results for duplicates, laboratory control samples, blank analysis, and interlaboratory comparison studies are provided within this section. Summary tables are also included.

DUPLICATES (PRECISION)

The duplicate sample results obtained for 2000 are summarized in Table 9.1. All analysis/matrix categories had 75 percent or better of their field duplicates fall within the established control limits except for ^{226}Ra in water and $^{239+240}\text{Pu}$ in water and air. With only three duplicates reported during the year for ^{226}Ra there are too few data points to come to any logical conclusions. However with 13 duplicate results being reported for $^{239+240}\text{Pu}$ in both air and water, a legitimate problem does appear to exist with this method routinely meeting the given control limits. Table 9.1 shows that only 7 of 13 duplicate results (54 percent) reported for the air matrix and 9 of 13 duplicate results (69 percent) reported for the water matrix are in control. This is perhaps a result of the uncertainties associated with the sample dissolution during chemical preparation and the counting efficiency of the alpha spectroscopy counting technique. The variance of air volumes and air pressures experienced during sample collection as well as the transfers of multiple air filters for screening and compositing could also be contributing factors to the poor duplicate precision observed for the air matrix. Wider acceptance windows should be investigated for use with the $^{239+240}\text{Pu}$ analysis for both the air and water matrices in the future.

LABORATORY CONTROL SAMPLES (ACCURACY)

The laboratory control sample (LCS) results obtained for 2000 are summarized in Table 9.2. The LCS results were satisfactory with no more than one result being out of control for any given analysis/matrix category for the year.

BLANK ANALYSIS

The laboratory blank sample results obtained for 2000 are summarized in Table 9.3. The laboratory blank results were satisfactory with no more than one result being out of control for any given analysis/matrix category for the year.

INTERLABORATORY COMPARISON STUDIES

The interlaboratory comparison sample results obtained for 2000 are summarized in Tables 9.4 through 9.6.

Table 9.4 shows the summary of interlaboratory comparison sample results for the BN in-house Analytical Services Laboratory (ASL). The ASL participated in the InterLaB RadChemTM Proficiency Testing Program directed by Environmental Resource Associates (ERA), the Quality Assessment Program (QAP) administered by the NNSA Environmental Measurements Laboratory (EML), the Radiochemistry Intercomparison Program provided by the National Institute of Standards and Technology (NIST), and the Mixed Analyte Performance Evaluation Program (MAPEP) conducted by the Idaho National Engineering and Environmental Laboratory (INEEL). The ASL performed very well during the year by passing 56 out of 58 parameters analyzed. The only outliers were two ^{65}Zn results analyzed by gamma spectroscopy for the ERA program. Both results were out of control with high bias.

Table 9.5 shows the summary of interlaboratory comparison sample results for the Subcontracted Radiochemistry Laboratory. The Subcontractor participated in the InterLaB RadChemTM Proficiency Testing Program directed by ERA, the QAP administered by EML, and the MAPEP conducted by INEEL. The Subcontractor performed very well during the year by passing 45 out of 48 parameters analyzed. Two of the outliers were ^{226}Ra results analyzed by radon emanation technique for the ERA program. One of these results was reported with high bias, while the other was reported with low bias. The laboratory was successful in passing two

other ERA performance evaluation rounds of ^{226}Ra during the year. The third outlier was ^{134}Cs , which was analyzed by gamma spectroscopy for the ERA program and was reported with low bias.

Table 9.6 shows the summary of interlaboratory comparison sample results for the BN in-house Dosimetry Group. They participated in the Battelle Pacific Northwest National Laboratory (PNNL) performance evaluation study program during the course of the year. The Dosimetry Group performed very well during the year by passing 17 out of 18 TLDs analyzed. The only outlier was a S60/Cf-252 UN. Mixture (1:3) within the test range of 0.03 to 5 rem.

9.5 ESTIMATES OF DATA QUALITY

The measurement quality as discussed in Section 9.3 indicate that representativeness, comparability and quality control of the data reported are acceptable. Further, data completeness for this data set met or exceeded completeness goals so these data are acceptable for their intended use.

Table 9.1 Summary of Field Duplicate Samples - 2000

Analysis	Number of Duplicate Matrix	Number of Results Reported	Number Within Control Limits ^(a)
Gross Alpha	Air	72	59
Gross Beta	Air	72	66
²³⁹⁺²⁴⁰ Pu	Air	13	7
Gamma	Air	29	25
Tritium	Air	36	32
Gross Alpha	Water	5	5
Gross Beta	Water	14	12
²³⁹⁺²⁴⁰ Pu	Water	13	9
Gamma	Water	33	26
Tritium	Water	44	41
⁹⁰ Sr	Water	6	6
²²⁶ Ra	Water	3	1
²²⁸ Ra	Water	3	3
TLDs	Ambient Radiation	380	363

- (a) Control limits are as follows: If the result is less than 5 times detection level, then duplicate results must be \pm detection level. If the result is greater than 5 times detection level, then results must be \pm 20 percent (Relative Percent Difference). The \pm 20 percent Relative Percent Difference is used as the control limit for all TLD duplicates.

Table 9.2 Summary of Laboratory Control Samples (LCS) - 2000

Analysis	Matrix	Number of LCS Results Reported	Number Within Control Limits ^(a)
Gross Alpha	Air	18	16
Gross Beta	Air	18	18
²³⁹⁺²⁴⁰ Pu	Air	8	8
Gamma	Air	30	30
Tritium	Air	12	11
Gross Alpha	Water	8	8
Gross Beta	Water	11	11
²³⁹⁺²⁴⁰ Pu	Water	6	6
Gamma	Water	34	34
Tritium	Water	23	22
⁹⁰ Sr	Water	5	4
²²⁶ Ra	Water	3	2
²²⁸ Ra	Water	3	2

(a) Control limits are as follows: 80 to 120 percent for all analyses and matrices except for gross alpha and beta which are 50 to 120 percent.

Table 9.3 Summary of Laboratory Blank Samples - 2000

Analysis	Matrix	Number of Blank Results Reported	Number Within Control Limits ^(a)
Gross Alpha	Air	36	36
Gross Beta	Air	36	35
²³⁹⁺²⁴⁰ Pu	Air	8	8
Gamma	Air	21	21
Tritium	Air	10	10
Gross Alpha	Water	8	8
Gross Beta	Water	11	11
²³⁹⁺²⁴⁰ Pu	Water	6	6
Gamma	Water	31	31
Tritium	Water	22	22
⁹⁰ Sr	Water	5	5
²²⁶ Ra	Water	3	3
²²⁸ Ra	Water	3	3

(a) Control limit is less than detection level.

Table 9.4 Summary of Interlaboratory Comparison Samples for the BN in-house Analytical Services Laboratory - 2000

Analysis	Matrix	Number of Results Reported	Number Within Control Limits ^(a)
<i>ERA Results</i>			
Gross Alpha	Water	4	4
Gross Beta	Water	4	4
Gamma	Water	16	14
Tritium	Water	1	1
⁹⁰ Sr	Water	3	3
²²⁶ Ra	Water	2	2
²²⁸ Ra	Water	2	2
<i>EML Results</i>			
Gross Alpha	Air	1	1
Gross Beta	Air	1	1
²³⁹⁺²⁴⁰ Pu	Air	1	1
Gamma	Air	5	5
Gross Alpha	Water	1	1
Gross Beta	Water	1	1
²³⁹⁺²⁴⁰ Pu	Water	1	1
Gamma	Water	3	3
Tritium	Water	1	1
⁹⁰ Sr	Water	1	1
<i>NIST Results</i>			
²³⁹⁺²⁴⁰ Pu	Air	1	1
⁹⁰ Sr	Air	1	1
<i>MAPEP Results</i>			
Gamma	Water	6	6
²³⁹⁺²⁴⁰ Pu	Water	1	1
⁹⁰ Sr	Water	1	1

(a) Control limits are determined by the individual interlaboratory comparison study.

Table 9.5 Summary of Interlaboratory Comparison Samples for the Subcontract Radiochemistry Laboratory - 2000

Analysis	Matrix	Number of Results Reported	Number Within Control Limits ^(a)
<i>ERA Results</i>			
Gross Alpha	Water	2	2
Gross Beta	Water	2	2
Gamma	Water	8	7
Tritium	Water	1	1
⁹⁰ Sr	Water	3	3
²²⁶ Ra	Water	4	2
²²⁸ Ra	Water	3	3
<i>EML Results</i>			
Gross Alpha	Air	1	1
Gross Beta	Air	1	1
²³⁹⁺²⁴⁰ Pu	Air	1	1
Gamma	Air	4	4
Gross Alpha	Water	1	1
Gross Beta	Water	1	1
²³⁹⁺²⁴⁰ Pu	Water	1	1
Gamma	Water	4	4
Tritium	Water	1	1
⁹⁰ Sr	Water	1	1
<i>MAPEP Results</i>			
Gamma	Water	7	7
²³⁹⁺²⁴⁰ Pu	Water	1	1
⁹⁰ Sr	Water	1	1

(a) Control limits are determined by the individual interlaboratory comparison study.

Table 9.6 Summary of Interlaboratory Comparison Thermoluminescent Dosimeter (TLD) Samples for the BN in-house Dosimetry Group - 2000

Analysis	Matrix	Number of Results Reported	Number Within Control Limits ^(a)
TLDs	Ambient Radiation	18	17

(a) Control limits are determined by the Battelle Pacific Northwest National Laboratory (PPNL) performance evaluation study program.



Shoshone Mountain Looking South of Mid Valley (No Date Provided)